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(54) **Method for evaporating metal using a resistance heated, pyrolytic boron nitrided coated graphite boat**

Verfahren zur Verdampfung von Metall mittels eines mit pytolytischem Bornitrid überzogenen Graphit-Schiffchens mit Widerstandsheizung

Procédé d'évaporation de métal utilisant une nacelle en graphite revêtue de nitrure de bore pyrolytique et chauffée par résistance

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- **INSTRUMENTS AND EXPERIMENTAL TECHNIQUES vol. 22, no. 1, 1979, NEW YORK US pages 253 - 255 SELIVERSTOV 'Production of films of substances with complicated composition by discrete-pulse evaporation' ***
- **PATENT ABSTRACTS OF JAPAN vol. 8, no. 222 (C-246)(1659) 9 October 1984 &**

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Description

[0001] The present invention relates to a method of evaporating a metal using a graphite container having a pyrolytic boron nitride coating.

[0002] Many metals such as aluminum, copper, chromium, zinc and tin are coated onto various substrates such as metal, glass and plastic by a vacuum deposition process in which a container is heated by electric resistance heating to vaporize metal fed into contact with the container. The container, which is commonly referred to as a "boat", is connected in an electric circuit in a series circuit relationship so that current flows directly through the boat, which in turn heats the metal in contact with the boat until it vaporizes. The metal is vaporized in an evacuated atmosphere for coating a product which may be individually introduced into the evacuated chamber or continuously fed through the chamber. Discreet products may include a television picture tube, an automobile head light, a toy or the like.

[0003] Presently, most resistance heated containers are composed of an intermetalic ceramic composite of titanium diboride and boron nitride alone or in combination with aluminum nitride. The composite has a very short lifetime and requires continual adjustment of the power supply. Moreover, the resistance characteristics of such heaters are not stable during operation since the metal component of the ceramic composite is a conductor which forms part of the electrical circuit. As a result metal vaporization is not uniform resulting in a non-uniform metal deposition.

[0004] A container of graphite coated with pyrolytic boron nitride has been suggested for use as an alternative to a resistance heater composed of an intermetallic ceramic composite. The coating of pyrolytic boron nitride is intended to electrically isolate the molten metal in the container from the current path through the graphite body and to supply more uniform heat to the metal. It has instead been discovered that the molten metal after only a short time interval will pass through the layer planes of the pyrolytic boron nitride coating and directly infiltrate the porous graphite body. Changes in the thermal cycle causes the boron nitride coating to crack within a short operating time interval. This destroys the utility of the graphite for use as a metal evaporator. Increasing the thickness of the pyrolytic boron nitride coating can delay, but will not significantly prevent leakage through the boron nitride coating.

[0005] L.A. Seliverstov in instruments and Experimental Techniques col. 22, No. 1 (1979) pp 253-5 describes a method for the production of films of substances with complicated composition, eg. a magnetic alloy, by discrete pulse evaporation. One benefit of the method is an increase in the lifetime of the evaporator because of the short time of contact with the evaporated material. JP-A-59-104 472 discloses a method of flash vapour deposition with a cycle in which the current supply to the evaporation boat is interrupted to.

[0006] According to the present invention there is provided a method of evaporating a metal using a graphite container having a graphite body and a pyrolytic boron nitride coating comprising the steps of: depositing a metal charge of predetermined weight into said container over said boron nitride coating; applying electrical power to said container such that current flows directly through the graphite body to cause the metal charge to fully evaporate within a finite time interval of less than two (2) minutes, varying the electrical power being applied to said container to control the heat cycle across said boron nitride coating and to provide a cool down period in each cycle of applied power, and introducing a new charge of metal into said container in a semi-continuous fashion upon completion of each heat cycle.

[0007] According to a further feature of the present invention there is provided a method of evaporating a metal using a graphite container having a graphite body with a cross sectional area of between 15 sq. mm and 72 sq.mm and a maximum length of 20.32cms (8 inches), and a pyrolytic boron nitride coating of between 0.025cms (.010 inches) and 0.05cms (.020. inches) thick, comprising the steps of: depositing a metal charge selected from the group consisting of aluminum, copper, zinc and tin with said metal charge having a predetermined weight of between 6 mg and 200 mg into said container with the boron nitride coating separating the graphite body from the metal charge; applying electrical power with a constant current and a variable voltage of between 4 to 25 volts to said graphite container for a controlled short interval of time of less than two (2) minutes sufficient to cause the metal charge to fully evaporate in said time interval and the current to flow directly through said graphite body; varying the electrical power applied to said container to control the heat cycle across said coating with said electrical power being interrupted repetitively to provide a minimum cool down period with essentially no current flow between each application of applied power and introducing a new charge of metal into said container during said cool down period.

[0008] An aim of the present invention is to provide a method of using a container of graphite coated with pyrolytic boron nitride for metal vaporization, which method improves performance.

[0009] In accordance with the method of the present invention a graphite container coated with boron nitride is used for vaporizing metal in contact with the container through heat conduction and will provide an extended service life if the metal to be vaporized is deposited in a semi-continuous fashion while varying the applied power to the container to provide a controlled heat cycle in which the applied power is applied over a fixed time period of less than two minutes sufficient to fully evaporate the metal charge, and to provide a cool down period before reintroducing a new metal charge.

[0010] The present invention will now be further described, by way of example, with reference to the accompanying drawings, in which:-

Fig. 1 is a perspective view of a graphite metal evaporator with a pyrolytic boron nitride coating for use in accordance with the present invention;

Figure 2 is a cross section taken along the lines 2-2 of Figure 1; and

Figure 3 is a plot of the current flow pattern through the evaporator of Figure 1 during the practice of the present invention.

[0011] A resistance heater 10 shown in Figure 1 comprises a graphite body 12 with a pyrolytic boron nitride coating 14 covering a specified area of the graphite body 12 in which a depression 15 is formed. The graphite body 12 is a mechanical block of high strength graphite. The depression 15 can be of any desired shape or geometry, preferably rounded as is shown in Figure 2 with hemispherical ends as shown in Figure 1, to receive and hold a metal charge (not shown) to be vaporized by the resistance heater 10. The metal charge may be composed of any metal selected from the group consisting of aluminum, copper, zinc and tin and may be in the form of one or more pieces having a total weight of preferably between 6 mg to 200 mg.

[0012] The graphite body 12 is preferably of a rectangular configuration having a predetermined cross sectional area to provide a defined resistance path which will optimize the heat generated therein for a given applied voltage, to assure complete evaporation of the metal charge under controlled heat cycle conditions as hereinafter discussed. The graphite body 12 must be properly sized to allow it to cause evaporation of the metal charge within the desired time interval. The applied voltage, cross sectional area and length of the graphite body 12 will determine the heat cycle time. To achieve full evaporation of the metal charge in less than two minutes with an applied voltage of from 4 to 25 volts it is necessary for the graphite body 12 to have a maximum cross sectional area of between about 15 mm^2) (.02 square inch) and 72mm^2 (.11 square inch) and a maximum length of 20.32cms (8 inches) although preferably less than 12.7cms (5 inches). An evaporator with a 15sq. mm cross section and a 200 mm length would require an applied voltage of 25 volts whereas a 72 sq. mm cross section and a 75 mm length requires an applied voltage of only 4 volts.

[0013] A coating of pyrolytic boron nitride 14 may be formed on the graphite body 12 by passing gaseous vapors of ammonia and boron halide e.g. boron trichloride as reactants into a heated reactor (not shown). The temperature of the reactor is elevated to a temperature of up to 2300°C at a pressure of less than 50 mm of mercury to form a coating, by chemical vapor deposition, of boron nitride as is taught in U.S. Patent No. 3,152,006. The pyrolytic boron nitride coating 14 must be thin enough to maximize heat transfer into the metal charge so as to permit evaporation within the desired fixed time interval of less than two minutes and yet must be thick enough to maintain its structural integrity over an extended service lifetime with constant heat cycle variations. The preferred thickness range for the pyrolytic boron nitride coating should be between 0.025cms and 0.05cms (.010 and .020 inches).

[0014] The service life of the heater 10 has proven to be orders of magnitude greater than conventional resistance heaters. The heater 10 is operated in a semi-continuous fashion with a metal charge fully evaporated during each heat cycle in a period of under two minutes. In each cycle of operation power is applied for a short interval coincident with the time required to fully evaporate the metal charge and includes a cool down period before reapplying power. This results in a current flow pattern as shown in Figure 3 in which the current is almost constant over the time period required to cause full evaporation of the metal charge followed by a cool down period in which a new metal charge is deposited before power is reapplied. By operating the heater 10 in this semi-continuous fashion the pyrolytic boron nitride coating 14 acts only as an insulating barrier with no leakage of metal through the boron nitride coating 14 into the graphite body 12.

Claims

1. A method of evaporating a metal using a graphite container (10) having a graphite body (12) and a pyrolytic boron nitride coating (14) comprising the steps of: depositing a metal charge of predetermined weight into said container (10) over said boron nitride coating (14); applying electrical power to said container (10) such that current flows directly through the graphite body (12) to cause the metal charge to fully evaporate within a finite time interval of less than two (2) minutes, varying the electrical power being applied to said container (10) to control the heat cycle across said boron nitride coating (14) and to provide a cool down period in each cycle of applied power, and introducing a new charge of metal into said container (10) in a semi-continuous fashion upon completion of each heat cycle.

2. A method of evaporating a metal using a graphite container (10) having a graphite body (12) with a cross sectional area of between 15 sq. mm and 72 sq.mm and a maximum length of 20.32cms (8 inches), and a pyrolytic boron nitride coating (14) of between 0.025cms and 0.05cms (.010 and .020 inches) thick, comprising the steps of: depositing a metal charge selected from the group consisting of aluminum, copper, zinc and tin with said metal charge having a predetermined weight of between 6 mg and 200 mg into said container (10) with the boron nitride coating (14) separating the graphite body (12) from the metal charge; applying electrical power with a constant current and a variable voltage of between 4 to 25 volts to said graphite container (10)

for a controlled short interval of time of less than two (2) minutes sufficient to cause the metal charge to fully evaporate in said time interval and the current to flow directly through said graphite body (12); varying the electrical power applied to said container (10) to control the heat cycle across said coating (14) with said electrical power being interrupted repetitively to provide a minimum cool down period with essentially no current flow between each application of applied power and introducing a new charge of metal into said container (10) during said cool down period.

Patentansprüche

1. Verfahren zur Verdampfung eines Metalls unter Verwendung eines Graphitbehälters (10) mit einem Graphitkörper (12) und einer pyrolytischen Bornitrid-Beschichtung (14), umfassend die Schritte: Anordnen einer Metallcharge vorbestimmten Gewichts in dem Behälter (10) über der Bornitrid-Beschichtung (14); Anlegen elektrischer Leistung an den Behälter (10), so daß der Strom direkt durch den Graphitkörper (12) fließt, so daß die Metallcharge innerhalb eines endlichen Zeitraums von weniger als zwei (2) Minuten vollständig verdampft, Ändern der an den Behälter (10) angelegten elektrischen Leistung zur Steuerung des Heizzyklus quer zur Bornitrid-Beschichtung (14) und zur Bereitstellung eines Abkühlzeitraums in jedem Zyklus der angelegten Leistung, und Einführen einer neuen Metallcharge in den Behälter (10) in halbkontinuierlicher Weise nach Ablauf jedes Heizzyklus.

2. Verfahren zur Verdampfung eines Metalls unter Verwendung eines Graphitbehälters (10), der einen Graphitkörper (12) mit einer Querschnittsfläche zwischen 15 mm^2 und 72 mm^2 und einer maximalen Länge von 20,32 cm (8 Inch) und eine pyrolytische Bornitrid-Beschichtung (14) zwischen 0,025 cm und 0,05 cm (0,010 und 0,020 Inch) Dicke aufweist, umfassend die Schritte: Anordnen einer Metallcharge, ausgewählt aus der Gruppe, bestehend aus Aluminium, Kupfer, Zink und Zinn, wobei die Metallcharge ein vorbestimmtes Gewicht zwischen 6 mg und 200 mg aufweist, in dem Behälter (10), wobei die mit der Bornitrid-Beschichtung (14) den Graphitkörper (12) von der Metallcharge trennt; Anlegen einer elektrischen Leistung mit einem Konstantstrom und einer veränderlichen Spannung zwischen 4 und 25 Volt an den Graphitbehälter (10) für einen gesteuerten kurzen Zeitraum von weniger als zwei (2) Minuten, ausreichend, um die Metallcharge in dem Zeitraum vollständig zu verdampfen, wobei der Strom direkt durch den Graphitkörper (12) fließt; Ändern der an den Behälter (10) angelegten elektrischen Leistung zur Steuerung des Heizzyklus quer zur Beschichtung (14), wobei die elektrische Leistung wiederholt unterbrochen wird, um einen minimalen Abkühlzeitraum, bei dem im wesentlichen kein Strom fließt, zwischen jedem Anlegen von Leistung bereitzustellen, und Einführen einer neuen Metallcharge in den Behälter (10) während des Abkühlzeitraums.

Revendications

1. Un procédé permettant d'évaporer un métal utilisant un récipient en graphite (10) possédant un corps en graphite (12) et un revêtement de nitrure de bore pyrolytique (14) comprenant les étapes suivantes : le dépôt d'une charge de métal d'un poids prédéterminé dans ledit récipient (10) sur ledit revêtement de nitrure de bore (14) ; l'application de l'énergie électrique audit récipient (10) de sorte que le courant passe directement à travers le corps en graphite (12) afin de provoquer l'évaporation totale de la charge de métal dans les limites d'un intervalle de temps donné dont la durée est inférieure à deux (2) minutes ; la variation de l'énergie électrique qui est appliquée audit récipient (10) afin de contrôler le cycle thermique sur ledit revêtement de nitrure de bore (14), et d'offrir une période de refroidissement pendant chaque cycle de la puissance appliquée, et l'introduction d'une nouvelle charge de métal dans ledit récipient (10) de façon semi-continue une fois que chaque cycle thermique est terminé.

2. Un procédé permettant d'évaporer un métal utilisant un récipient en graphite (10) possédant un corps en graphite (12) présentant une coupe transversale entre 15 mm^2 et 72 mm^2 et une longueur maximale de 20,32 cm (8 pouces), et un revêtement de nitrure de bore pyrolytique (14) dont l'épaisseur se situe entre 0,025 cm et 0,05 cm (entre 0.010 et 0.020 pouce), comprenant les étapes suivantes : le dépôt d'une charge de métal sélectionnée à partir d'un groupe composé d'aluminium, de cuivre, de zinc et d'étain, la charge de métal ayant un poids prédéterminé situé entre 6 mg et 200 mg dans ledit récipient (10) alors que le revêtement de nitrure de bore (14) constitue une séparation entre le corps en graphite (12) et la charge de métal ; l'application de l'énergie électrique, composée d'un courant constant et d'une tension variable située entre 4 et 25 Volts, audit récipient en graphite (10) pendant un bref intervalle de temps contrôlé d'une durée inférieure à deux (2) minutes qui est suffisante pour provoquer l'évaporation totale de la charge de métal dans les limites dudit intervalle de temps, et pour faire passer directement le courant à travers ledit corps en graphite (12) ; la variation de l'énergie électrique appliquée audit récipient (10) afin de contrôler le cycle thermique sur ledit revêtement (14)

alors que ladite énergie électrique est interrompue de façon répétée afin d'offrir une période de refroidissement minimum, en ce sens que fondamentalement aucun flux ne passe entre chaque application de la puissance appliquée, et l'introduction d'une nouvelle charge de métal dans ledit récipient (10) pendant ladite période de refroidissement.

Fig. 1

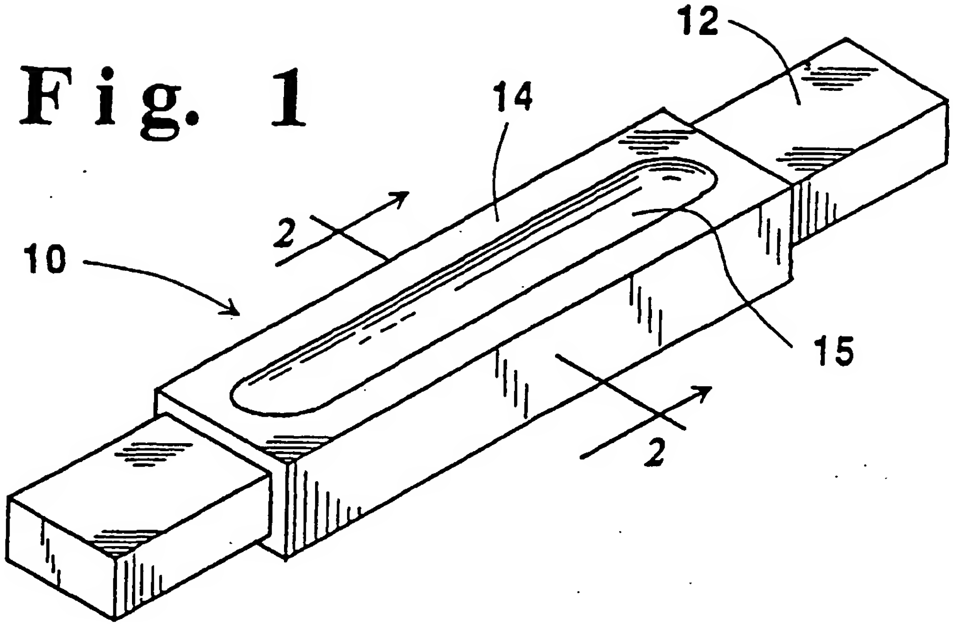
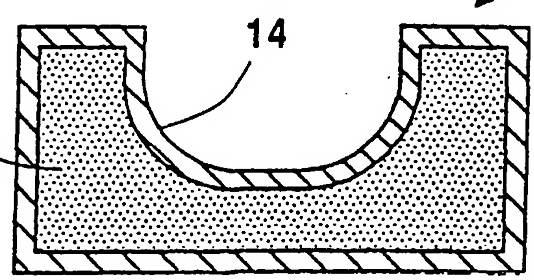


Fig. 2



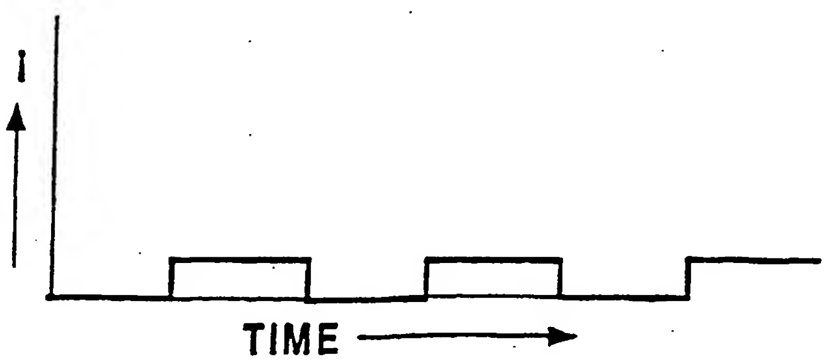


Fig. 3